

CLAIMS

1. A process for the solid phase continuous polymerisation of polyesters, comprising the steps of:
 - preparing a mass of granules, crystallised and at high temperature, of at least a
 - 5 polyester;
 - feeding said granules into a plant comprising at least an horizontal, cylindrical, rotary reactor (15), said reactor being slightly inclined;
 - producing a purge gas flow inside said reactor (15);
 - causing the intrinsic viscosity (IV) increase of said at least one polyester by making
 - 10 said granules move forward through said reactor (15) thanks to its rotation and inclination.
2. A process according to claim 1, wherein downstream said reactor (15) at least a second horizontal, cylindrical, rotary, slightly inclined reactor is provided.
3. A process according to claim 2, wherein said granules move from one reactor to the subsequent one by gravity.
- 15 4. A process according to claim 2, wherein the temperature of the polyester granules, subjected to the polymerisation passing through said reactors, is increased during the movement from one reactor to the subsequent of a value comprised between 2 and 20°C.
5. A process according to claim 2, wherein the temperature of the polyester granules passing through said reactors is decreased from one reactor to the subsequent of a value
- 20 comprised between 2 and 10°C.
6. A process according to claim 4, wherein the temperature of the polyester granules exiting one reactor is increased by means of an intermediate pre-heater located before the entrance into the subsequent reactor.
7. A process according to claim 2, wherein the polymerisation temperature in a first
- 25 reactor is lower than the polymerisation temperature in a second reactor located downstream

said first reactor.

8. A process according to claim 2, wherein the temperature of the polyester granules exiting one reactor is decreased by means of an intermediate cooler located before the entrance into the subsequent reactor.
- 5 9. A process according to any claim from 1 to 8, wherein the crystallisation degree X_c of said polyester granules fed into said reactor is comprised in the range of 0 + 70%.
10. A process according to any claim from 1 to 8, wherein said polyester granules fed into said reactor have a crystallisation degree $X_c > 10\%$.
11. A process according to any claim from 1 to 8, wherein said polyester granules fed into
10 said reactor have a crystallisation degree $X_c > 20\%$.
12. A process according to any claim from 1 to 8, wherein said polyester granules fed into said reactor have a crystallisation degree X_c comprised in the range of 0 + 50%.
13. A process according to any claim from 1 to 12, wherein said polyester granules fed into said reactor have a temperature comprised in the range of 170 – 235°C.
- 15 14. A process according to any claim from 1 to 12, wherein said polyester granules fed into said reactor have a temperature comprised in the range of 185 – 225°C.
15. A process according to any claim from 1 to 12, wherein said polyester granules fed into said reactor have a temperature comprised in the range of 180 – 230°C.
16. A process according to any preceding claim, wherein the rotation of said reactor occurs
20 around its own central axis (S).
17. A process according to any preceding claim, wherein said reactor (15) rotates at a speed comprised between 0,1 and 10 r.p.m..
18. A process according to any claim from 1 to 16, wherein said reactor rotates at a speed comprised between 0,1 and 2,0 r.p.m..
- 25 19. A process according to any claim from 1 to 18, wherein the angle of inclination (α) of

said reactor with respect to the horizontal plane is comprised between 3,0 and 12,0°.

20. A process according to any claim from 1 to 18, wherein the angle of inclination (α) of said reactor with respect to the horizontal plane is comprised between 0,1 and 3,5°.

21. A process according to any claim from 1 to 20, wherein the ratio between
5 the length of said reactor and its diameter of reactor is > 5 .

22. A process according to any preceding claim, wherein the ratio between the residual volume in said reactor inside which said granules have been fed, and its unloaded volume is $> 0,1$.

23. A process according to any preceding claim, wherein the flow regime of the polyester
10 granules inside the reactor is characterised by a Froude Number $Fr = (\omega^2 \cdot R/g)$ comprised in the range of $1 \cdot 10^{-4} \div 0,5$, where ω is the angular velocity of the reactor; R is the internal radius of the reactor and g is the gravity acceleration = 9,806 m/s².

24. A process according to any preceding claim, wherein the temperature inside said at least one reactor is maintained at a constant value $\pm 10^\circ\text{C}$.

15 25. A process according to any preceding claim, wherein the internal diameter of said reactor is comprised between 0,5 and 10 meters.

26. A process according to any claim from 1 to 24, wherein the internal diameter of said reactor is comprised between 0,3 and 6 meters.

27. A process according to any preceding claim, wherein the flow of said purge gas in said
20 reactor is conveyed in an opposite direction with respect to the flow direction of said granules that pass through said reactor.

28. A process according to any claim from 1 to 26, wherein the flow of said purge gas in said reactor is conveyed in the same direction with respect to the flow direction of said granules that pass through said reactor.

25 29. A process according to any preceding claim, wherein the ratio between the mass of the

purge gas flow that passes through said reactor and the mass of the polyester granules in the reactor is $> 0,62$.

30. A process according to any preceding claim, wherein the ratio between the mass of the purge gas flow that passes through said reactor and the mass of the polyester granules in the
5 reactor is $> 0,9$.

31. A process according to any preceding claim, wherein said purge gas is an inert gas or air.

32. A process according to any claim from 1 to 30, wherein said purge gas is air with a dew point $< - 30^{\circ}\text{C}$.

10 33. A process according to any claim from 1 to 30, wherein said purge gas is a mixture of gases chosen from the group comprising nitrogen, noble gases, carbon dioxide, carbon monoxide and oxygen and wherein the oxygen content is $< 10\%$ by weight.

34. A process according to any claim from 1 to 30, wherein said purge gas is a mixture of gases chosen from the group comprising nitrogen, noble gases, carbon dioxide, carbon
15 monoxide and oxygen and wherein the oxygen content is $< 6\%$ by weight.

35. A process according to any preceding claim, wherein the purge gas is recycled to the reactor, after having been purified of the organic impurities, until a level of organic impurities ≤ 100 p.p.m. by weight (CH_4 equivalent) has been reached.

36. A process according to any preceding claim, wherein said polyester is polyethylene
20 terephthalate or PET.

37. A process according to claim 36, wherein said polyethylene terephthalate or PET has an IPA (Isophthalic Acid) content comprised in the range of $1 \pm 20\%$.

38. A process according to claim 36 or 37, wherein said granules of polyethylene terephthalate fed into said reactor have an intrinsic viscosity comprised in the range between
25 0.55 and 0.65 dl/g.

39. A process according to claim 36 or 37, wherein said granules of polyethylene terephthalate fed into said reactor have an intrinsic viscosity comprised in the range between 0.25 and 0.75 dl/g.
40. A process according to any claim from 1 to 35, wherein said polyester is PEN
5 polyethylene naphthalate.
41. A process according to any claim from 1 to 35, wherein said polyester is PBT polybutylene terephthalate.
42. A process according to any claim from 1 to 35, wherein said granules fed in the reactor have a carboxyl end groups content in the range of 10 ÷ 45%.
- 10 43. A process according to any preceding claim, wherein said granules are cube-shaped with dimensions comprised between 1 mm³ and 125 mm³.
44. A process according to any claim from 1 to 42, wherein said granules are spherical with a diameter comprised between 1 mm and 5 mm.
45. A process according to any claim from 1 to 42, wherein said granules are extended
15 cylinders of length < 10 mm and circular or square cross-section having, respectively, diameter and side < 5 mm.
46. A process according to any claim from 1 to 42, wherein said polyester granules are pancake-like platelets of diameter > 3 mm and thickness < 3 mm.
47. A process according to any claim from 1 to 42, wherein said polyester granules have
20 an irregular shape with a volume comprised between 1 and 125 mm³.
48. A process according to any preceding claim, wherein said mass of crystallised granules is achieved by subjecting the polyester granules to a crystallisation step in a fluidised-bed crystalliser (13), said bed being fluidised by means of a gas flow sufficient to generate the fluidisation of the polyester granules with or without mechanical vibration.
- 25 49. A process according to claim 48, wherein said gases employed for the crystallisation

are inert gases or air.

50. A process according to claim 48 or 49, wherein said crystallisation step is performed with a residence time comprised between 2 and 20 minutes and, preferably, from 10 to 15 minutes.

5 51. A process according to claim 48, wherein the granules are heated to cause the crystallisation up to temperatures comprised between 140°C and 235°C and preferably in the range 200 – 225°C.

52. A process according to any preceding claim, wherein inside said reactor the polyester granules are subjected to a solid phase polycondensation and/or drying and/or crystallisation
10 and/or dealdehydisation.

53. A process according to any preceding claim, wherein the intrinsic viscosity of the polyester is increased of at least 0,35 dl/g during the solid phase polymerisation.

54. A process according to any preceding claim, wherein the intrinsic viscosity of the polyethylene terephthalate is increased of at least 0,4 dl/g during the solid phase
15 polymerisation.

55. A process according to any preceding claim, wherein said plant further comprises at least one vertical reactor located upstream and/or downstream said horizontal reactor (15).

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